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6,6′-Dimethoxy-2,2′-[*m*-phenylenedi(iminomethylene)]diphenol

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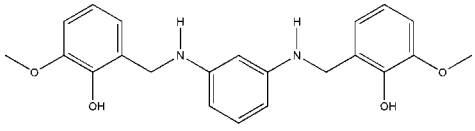
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.042; wR factor = 0.119; data-to-parameter ratio = 7.3.

In the title compound, $C_{22}H_{24}N_2O_4$, the molecules are linked into sheets by O—H···O and C—H···π hydrogen bonds, leading to fused $R_2^2(10)$ rings which form sheets parallel to the (001) plane. A crystallographic twofold rotation axis passes through the central benzene ring.

Related literature

For related literature, see: Allen *et al.* (1987); Bernstein *et al.* (1995); Xia *et al.* (2007).



Experimental

Crystal data

$C_{22}H_{24}N_2O_4$
M_r = 380.43
Orthorhombic, *Pnc*2
a = 13.2402 (18) Å
b = 7.7416 (14) Å
c = 9.7770 (12) Å
V = 1002.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.46 × 0.29 × 0.15 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.961, T_{max} = 0.987
4760 measured reflections
948 independent reflections
587 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.119
S = 1.05
948 reflections
129 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of ring C9–C12/C10*A*/C11*A*.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*A*···O2	0.82	2.24	2.687 (4)	115
O1—H1*A*···O2^{i}	0.82	2.05	2.811 (4)	153
C6—H6···*Cg*1^{ii}	0.93	2.70	3.579 (6)	157

Symmetry code: (i) $-x+2, -y+2, z$; (ii) $x, y+1, z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2307).

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supplementary materials

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6,6'-Dimethoxy-2,2'-[*m*-phenylenedi(iminomethylene)]diphenol

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Comment

As part of our investigation of crystal structure of *o*-vanillin diamine derivatives, we report here the crystal structure of a diamine derivative, *N*,*N*'-(2-hydroxy-3-methoxy benzyl) benzene-1,3-diamine, (I).

The asymmetric unit consists of one half-molecule and the molecule has a twofold axis on the atoms C9—C12 line, Fig. (1). The dihedral angle between the neighbouring benzen rings in the molecule is 77.95(0.11) °. Its bond lengths and angles are in agreement with that of similar compounds (Xia *et al.*, 2007; 2007*a*, *b*, *c*).) and are normal (Allen *et al.*, 1987). The most closely related compound is *N*,*N*'-(2-hydroxy-3-methoxy benzyl) benzene-1,4-diamine, (II) (Xia *et al.*, 2007). The principal difference between (I) and (II) concerns the intermoleculae aggregation. In (I), The molecules are linked into sheets involving a $R_2^2(10)$ rings (Bernstein *et al.*, 1995) through O—H···O and C—H···π hydrogen bonds (Fig. 2). Atoms O1 in the molecule (*x*, *y*, *z*) and O2 in the molecule (2 − *x*, 2 − *y*, *z*) act as hydrogen-bond donors to atoms O2 in the molecule (2 − *x*, 2 − *y*, *z*) and O1 in the molecule (*x*, *y*, *z*), respectively, in addition, atom C6 in the molecule (*x*, *y*, *z*) act as hydrogen-bond donor to central aryl ring of the molecule at (*x*, 1 + *y*, *z*), forming a sheet parallel to the [001] plane and there are no direction-specific interactions between adjacent sheets. By contrast, in (II), the molecule are linked into sheets by means of N—H···O and O—H···N hydrogen-bonds; the latter are absent from the structure of (I).

Experimental

Solutions of *N*,*N*'-bis(2-hydroxy-3-methoxy benzylene)benzene-1,4-diamine (10 mmol) in methanol-chloroform (*v*/*v* = 1/1) (20 ml) and $NaBH_4$ (40 mmol) were mixed, the mixture solution was stirred under room temperature for 30 h and then mixtures was filtered, and then solution was left to produce crystals of (I) slowly.

Refinement

All H atoms were located in difference Fourier maps. H atoms bonded to C, O and N atoms were treated as riding atoms, with C—H distances of 0.93 Å (aryl), 0.96 Å (methyl), 0.97 Å (methylene), O—H distances of 0.82 Å (hydroxy) and N—H distances of 0.86 Å (amino), U_{iso}(H) = 1.2 (aryl, methylene, amino) or 1.5 U_{eq}(C) (methyl or hydroxy).

Figures

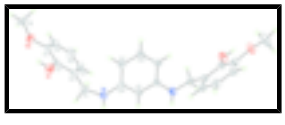

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are at the 30% probability level. Unlabelled atoms in the molecular are related to labelled atoms by 1 − *x*, 1 − *y*, *z*.

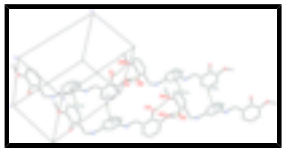

Fig. 2. A larger portion of the crystal structure of (I), showing the formation of a hydrogen-bonded sheet built from O—H···O and C—H···π. For clarity, H atoms not involved in the hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (A) *x*, 1 + *y*, *z*; (B) 2 − *x*, 2 − *y*, *z*; (C) 2 − *x*, 3 − *y*, *z*; (D) 1 − *x*, 2 − *y*, *z*; (E) 1 + *x*, 1 + *y*, *z*; (F) 1 + *x*, 2 + *y*, *z*].

6,6'-Dimethoxy-2,2'-[*m*-phenylenedi(iminomethylene)]diphenol

Crystal data

$C_{22}H_{24}N_2O_4$	F_{000} = 404
M_r = 380.43	D_x = 1.261 Mg m^{-3}
Orthorhombic, *Pnc*2	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2 -2bc	Cell parameters from 771 reflections
a = 13.2402 (18) Å	θ = 3.1–19.2º
b = 7.7416 (14) Å	μ = 0.09 mm^{-1}
c = 9.7770 (12) Å	*T* = 298 (2) K
V = 1002.1 (3) Å^3	Acerate, red
Z = 2	0.46 × 0.29 × 0.15 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	948 independent reflections
Radiation source: fine-focus sealed tube	587 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.064
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −15→12
T_{min} = 0.961, T_{max} = 0.987	*k* = −9→9
4760 measured reflections	*l* = −11→9

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.042	$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.0331P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.119	$(\Delta/\sigma)_{max} < 0.001$
S = 1.05	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
948 reflections	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
129 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 948 Freidel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 10 (10)

Secondary atom site location: difference Fourier map

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.6614 (3)	0.6391 (5)	0.8827 (4)	0.0504 (11)
H1	0.6551	0.6488	0.9699	0.060*
O1	0.9095 (2)	0.8177 (4)	0.6588 (4)	0.0574 (10)
H1A	0.9520	0.8633	0.6097	0.086*
O2	0.9070 (2)	1.1194 (4)	0.5231 (4)	0.0586 (10)
C1	0.7547 (3)	0.6954 (6)	0.8189 (6)	0.0511 (13)
H1B	0.7747	0.6104	0.7513	0.061*
H1C	0.8072	0.6998	0.8880	0.061*
C2	0.7477 (3)	0.8702 (6)	0.7503 (5)	0.0383 (11)
C3	0.8274 (3)	0.9240 (6)	0.6701 (5)	0.0385 (11)
C4	0.8230 (3)	1.0816 (6)	0.6010 (5)	0.0410 (12)
C5	0.7399 (4)	1.1859 (6)	0.6149 (5)	0.0524 (15)
H5	0.7370	1.2918	0.5701	0.063*
C6	0.6608 (4)	1.1320 (6)	0.6958 (6)	0.0578 (15)
H6	0.6042	1.2022	0.7054	0.069*
C7	0.6643 (3)	0.9769 (6)	0.7621 (5)	0.0503 (13)
H7	0.6099	0.9426	0.8158	0.060*
C8	0.9002 (4)	1.2617 (13)	0.4308 (8)	0.105 (3)
H8A	0.8922	1.3668	0.4817	0.157*
H8B	0.8431	1.2460	0.3716	0.157*
H8C	0.9607	1.2680	0.3769	0.157*
C9	0.5000	0.5000	0.8780 (6)	0.0382 (16)
H9	0.5000	0.5000	0.9731	0.046*
C10	0.5824 (3)	0.5705 (5)	0.8084 (5)	0.0361 (11)
C11	0.5805 (3)	0.5714 (5)	0.6655 (5)	0.0437 (12)
H11	0.6336	0.6202	0.6167	0.052*
C12	0.5000	0.5000	0.5972 (7)	0.0446 (17)
H12	0.5000	0.5000	0.5021	0.054*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.049 (2)	0.067 (3)	0.035 (2)	−0.018 (2)	−0.006 (2)	0.009 (2)
O1	0.0359 (17)	0.063 (2)	0.074 (2)	0.0088 (16)	0.0054 (19)	0.020 (2)
O2	0.041 (2)	0.072 (2)	0.063 (2)	−0.0089 (18)	0.0030 (19)	0.026 (2)
C1	0.039 (3)	0.062 (3)	0.053 (3)	−0.009 (2)	−0.003 (3)	0.011 (3)
C2	0.037 (2)	0.043 (3)	0.035 (2)	−0.004 (2)	−0.004 (2)	0.002 (2)
C3	0.033 (3)	0.045 (3)	0.037 (3)	−0.003 (2)	−0.002 (2)	0.002 (3)
C4	0.037 (3)	0.052 (3)	0.034 (3)	−0.008 (2)	−0.005 (2)	0.003 (3)
C5	0.056 (3)	0.042 (3)	0.059 (4)	−0.001 (2)	−0.007 (3)	0.005 (3)
C6	0.052 (3)	0.049 (3)	0.072 (4)	0.010 (2)	0.000 (3)	−0.006 (3)
C7	0.040 (3)	0.053 (3)	0.058 (4)	−0.003 (2)	0.007 (3)	−0.003 (3)
C8	0.082 (4)	0.138 (6)	0.094 (6)	−0.005 (4)	0.006 (4)	0.082 (5)
C9	0.045 (4)	0.043 (4)	0.026 (4)	−0.003 (3)	0.000	0.000
C10	0.038 (3)	0.036 (3)	0.035 (3)	−0.004 (2)	−0.002 (2)	0.003 (2)
C11	0.054 (3)	0.044 (3)	0.033 (3)	−0.004 (2)	0.007 (3)	0.007 (2)
C12	0.060 (4)	0.050 (4)	0.024 (3)	−0.004 (4)	0.000	0.000

Geometric parameters (Å, °)

N1—C10	1.380 (5)	C5—H5	0.9300
N1—C1	1.450 (6)	C6—C7	1.365 (7)
N1—H1	0.8600	C6—H6	0.9300
O1—C3	1.368 (5)	C7—H7	0.9300
O1—H1A	0.8200	C8—H8A	0.9600
O2—C4	1.380 (5)	C8—H8B	0.9600
O2—C8	1.427 (8)	C8—H8C	0.9600
C1—C2	1.513 (6)	C9—C10^{i}	1.397 (5)
C1—H1B	0.9700	C9—C10	1.397 (5)
C1—H1C	0.9700	C9—H9	0.9300
C2—C3	1.380 (6)	C10—C11	1.398 (6)
C2—C7	1.384 (6)	C11—C12	1.373 (6)
C3—C4	1.396 (6)	C11—H11	0.9300
C4—C5	1.371 (6)	C12—C11^{i}	1.373 (6)
C5—C6	1.377 (6)	C12—H12	0.9300
C10—N1—C1	122.3 (4)	C7—C6—H6	119.6
C10—N1—H1	118.8	C5—C6—H6	119.6
C1—N1—H1	118.8	C6—C7—C2	120.9 (4)
C3—O1—H1A	109.5	C6—C7—H7	119.6
C4—O2—C8	117.5 (4)	C2—C7—H7	119.6
N1—C1—C2	114.0 (4)	O2—C8—H8A	109.5
N1—C1—H1B	108.7	O2—C8—H8B	109.5
C2—C1—H1B	108.7	H8A—C8—H8B	109.5
N1—C1—H1C	108.7	O2—C8—H8C	109.5
C2—C1—H1C	108.7	H8A—C8—H8C	109.5
H1B—C1—H1C	107.6	H8B—C8—H8C	109.5

C3—C2—C7	118.5 (4)	C10^{i}—C9—C10	121.7 (6)
C3—C2—C1	118.4 (4)	C10^{i}—C9—H9	119.2
C7—C2—C1	123.1 (4)	C10—C9—H9	119.2
O1—C3—C2	118.2 (4)	N1—C10—C9	119.1 (4)
O1—C3—C4	121.4 (4)	N1—C10—C11	122.6 (4)
C2—C3—C4	120.4 (4)	C9—C10—C11	118.4 (4)
C5—C4—O2	125.2 (4)	C12—C11—C10	119.9 (5)
C5—C4—C3	120.1 (4)	C12—C11—H11	120.1
O2—C4—C3	114.7 (4)	C10—C11—H11	120.1
C4—C5—C6	119.2 (4)	C11—C12—C11^{i}	121.9 (6)
C4—C5—H5	120.4	C11—C12—H12	119.1
C6—C5—H5	120.4	C11^{i}—C12—H12	119.1
C7—C6—C5	120.9 (4)		
C10—N1—C1—C2	78.5 (5)	O2—C4—C5—C6	179.8 (4)
N1—C1—C2—C3	−170.8 (4)	C3—C4—C5—C6	−1.0 (7)
N1—C1—C2—C7	7.9 (7)	C4—C5—C6—C7	0.1 (7)
C7—C2—C3—O1	−179.9 (4)	C5—C6—C7—C2	0.4 (7)
C1—C2—C3—O1	−1.2 (6)	C3—C2—C7—C6	0.1 (7)
C7—C2—C3—C4	−0.9 (7)	C1—C2—C7—C6	−178.6 (4)
C1—C2—C3—C4	177.8 (4)	C1—N1—C10—C9	172.3 (4)
C8—O2—C4—C5	−11.9 (7)	C1—N1—C10—C11	−8.8 (6)
C8—O2—C4—C3	168.8 (5)	C10^{i}—C9—C10—N1	179.7 (4)
O1—C3—C4—C5	−179.6 (5)	C10^{i}—C9—C10—C11	0.8 (3)
C2—C3—C4—C5	1.4 (6)	N1—C10—C11—C12	179.5 (3)
O1—C3—C4—O2	−0.3 (6)	C9—C10—C11—C12	−1.6 (6)
C2—C3—C4—O2	−179.3 (4)	C10—C11—C12—C11^{i}	0.8 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···O2	0.82	2.24	2.687 (4)	115
O1—H1A···O2^{ii}	0.82	2.05	2.811 (4)	153

Symmetry codes: (ii) −*x*+2, −*y*+2, *z*.

Fig. 1

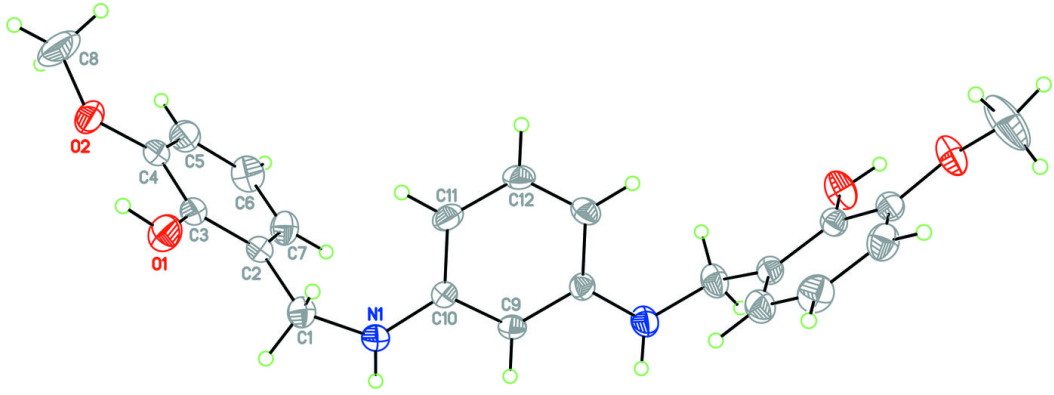

Fig. 2